

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.: 09/775,106
Filing Date: February 1, 2001
Applicant: Gerard A. Mourou
Group Art Unit: 1725
Examiner: Geoffrey S. Evans
Title: METHOD FOR CONTROLLING CONFIGURATION OF
LASER INDUCED BREAKDOWN AND ABLATION
Attorney Docket: 2115D-000939/DVC (UofM Ref. No. 939rld1)

Best Available Copy

DECLARATION OF JOHN NEES SUBMITTED
UNDER 37 C.F.R. § 1.132

I, John Nees hereby state and declare as follows:

1. I received my Bachelor of Science in Physics from Kansas State University and a Master of Science in Optics from the University of Rochester in Rochester, New York.
2. I have been employed by the University of Michigan as a research scientist for ultra fast optics at the University of Michigan Center for Ultra Fast Optical Science since 1988. I have been named as a joint inventor on several patents, I am the co-author in over 30 publications and I have been a presenter or joint presenter at several conferences; my curriculum vitae is attached. (Attachment I)
3. I have read and understood USPN 5,656,186, the Office Action dated April 12, 2005 and the Response to the Office Action being submitted herewith.

4. It is my understanding that the patent examiner has rejected certain claims in the subject application on the basis of Ihlemann et al. in the article "Nanosecond and Femtosecond Excimer Laser Ablation of Fused Silica." It is also my understanding that the patent examiner has rejected certain claims in the subject application on the basis of Alexander 6,489,589.

5. I have read and understood the basis of the Office Action rejections based on, respectively, Ihlemann & Alexander. I have read and understood the Ihlemann paper cited above and the Alexander patent cited above.

6. For the reasons given below, Ihlemann does not show:

(a) fluence at breakdown threshold, essentially accurate breakdown or any other related features found in the subject application;

(b) the point at which the size of the feature is not limited by thermal diffusion;

(c) ways of damage where the affected area is substantially determined by the beam shape and fluence in relation to the threshold for laser induced breakdown; or

(d) machining at ablation levels having anything to do with relating pulse width to breakdown threshold fluence.

7. For the reasons given below, Alexander does not show:

(a) fluence at breakdown threshold or essentially accurate breakdown;

(b) the point at which the size of the feature is not limited by thermal diffusion;

(c) the affected area substantially determined solely by the beam shape and fluence in relation to the threshold for laser induced breakdown;

(d) material being removed without any significant heat transfer; or

(e) machining without significant heat transfer to the surrounding areas of the work piece so that the surrounding areas are not significantly affected.

8. The rejection assumes that Ihlemann's Figure 1 shows making holes in SiO₂ with 500 femtosecond pulses and that Ihlemann's breakdown at 500 femtosecond pulse width must be essentially accurate.

9. Ihlemann, et al report a series of experiments using a number of lasers where the material removal rates are recorded as a function of laser fluence. See Figure 1 of Ihlemann. (Attachment II)

10. Ihlemann contrasts gentle etching at low material removal rate with explosive sputtering at high material removal rate. Ihlemann's graphical representations show microns per pulse, indicating the amount of material removed, corresponding to depth thereof.

11. These material removal rates are demonstrated for multiple pulses delivered after the material has already been exposed to conditioning pulses, typically 20 to 40 such pulses.

12. Ihlemann's mentioning of the term "gentle" only pertains to material removal rate and only after the surface has been preconditioned by delivery of a sequence of earlier pulses. Ihlemann at Figure 1 and page 367 make it clear that the conditions of "gentle etching" and "explosive sputtering" depend on amount of material removed and do not depend on pulse width.

13. There is no difference between the 22 nanosecond data and the 500 femtosecond data of Ihlemann.

14. There is no reference to accurate machining in Ihlemann.

15. Ihlemann does not identify a threshold energy for material removal at all. Ihlemann never varies pulse duration. Ihlemann never recognizes there is a breakpoint because he doesn't relate breakdown threshold fluence to pulse duration.

16. Ihlemann's failure to show a relationship between pulse duration and breakdown threshold fluence is evidenced by Figure 1 that contains a graph of data represented by diamonds and squares. The diamond curve data is for pulse duration of 500 femtoseconds (248 nm). The square curve data is for pulse duration of 22 nanoseconds (193 nm). (Attachment II)

17. In Ihlemann, there is no difference between the performance curve of 500 femtosecond pulses and 22 nanosecond pulses. The two curves coincide. Thus, Ihlemann makes no distinction between the 500 femtosecond pulses and the 22 nanosecond pulses performance in ablation (material removal) rate as a function of fluence. (Attachment II)

18. It is impermissible to overlook the importance of the relationship of fluence breakdown threshold and laser pulse width of the present Mourou invention of 5,656,186.

19. It is impermissible to interpret Ihlemann as teaching any features of the Mourou 5,656,186.

20. The rejection says that Alexander shows laser machining of stainless steel, gold, copper, iron, nickel, titanium, silicone and diamond using pulse width of 150 femtosecond duration and it is alleged and assumed that Alexander's machining is essentially accurate. The rejection based on Alexander is similar to the rejection based on Ihlemann.

21. The rejection assumes that Alexander is capable of material removal in a manner that is essentially accurate on the basis of the extent of heat transfer to surrounding areas.

22. Alexander is completely devoid of any teachings relevant to the present invention and is contrary to the present invention, at least because the conditions and teachings of Alexander lead to significant collateral damage during laser machining as is demonstrated by my comparable experiments as described herein below.

23. In Alexander at **Column 9, lines 30-37**, Alexander gives the following parameters: a $3\mu\text{m}$ spot size ($7 \times 10^{-8} \text{ cm}^2$), a pulse duration of 150fs, and a pulse energy of 50mJ. This produces a fluence of energy/area equal to $\sim 7 \times 10^5 \text{ J/cm}^2$. This fluence is five orders of magnitude over the damage threshold of the material.

24. I have conducted experiments for damage in glass induced at a fluence of 10^5 J/cm^2 with the spacing between damage spots of 40 microns. (See Figure A, Attachment III.)

25. Fig. A shows my experimental results where damage was induced is by a femtosecond laser at a fluence of only $1 \times 10^5 \text{ J/cm}^2$ with 1.1 mJ in a $1.2 \mu\text{m}$ spot. My experimental conditions are essentially identical to Alexander's. In both my experiments and Alexander's, the intensity was about 10^{18} W/cm^2 and x-rays (ionizing radiation) were produced. In my experiments, the damage sites are spaced by $40\mu\text{m}$, enabling one to see that physical damage and cracking extend tens of microns from the central location of the damage.

26. This level of irradiation per Alexander is preposterously high for machining applications, especially when minimal collateral damage is desired.

27. Clearly, the parameters given in Alexander lead to significant collateral damage.

28. Alexander states that shielding should be employed, as x-rays will be produced. This is because Alexander causes so much collateral damage.

29. Alexander does not teach that the shape of the affected area is substantially determined solely by the beam shape and fluence near the breakdown threshold fluence.

30. Alexander's parameters so far exceed the threshold that it is impossible to achieve or come close to determining breakdown threshold.

31. In contrast, Mouoru 5,656,186 clearly demonstrates and teaches that near the breakdown threshold fluence, the shape of the affected area is substantially determined solely by the beam shape and fluence. See Mouoru, **Column 5 lines 62 to 64**.

32. Alexander is concerned only with the rate at which pulses are generated. That is, pulse repetition rate. Alexander's repetition rate is the key feature of all of Alexander's claims.

33. Alexander is essentially devoid of useful teachings since Alexander's **Columns 2-9** describes what Alexander admits is background physics. The exemplary embodiments of Alexander beginning at **Column 9** reveal very little except for the preposterously high levels of energy and the importance of the repetition rate as disclosed in Alexander's claims.

34. In summary, it bears repeating that:

- a. Ihlemenn is concerned only with material removal rate, relating gentle etch to low material removal rate and sputtering to high material removal rate.
- b. Ihlemann and Alexander do not recognize a relationship between fluence breakdown threshold and pulse width.
- c. Ihlemann states there is no difference between a 500 femtosecond pulse and a 22 nanosecond pulse, as evidenced by his own data in Figure 1.
- d. Ihlemann and Alexander ignore that fluence is coupled with pulse width in order to achieve essentially accurate breakdown.
- e. There are no error bars in Ihlemann or Alexander showing any regime of increased accuracy.
- f. Alexander is only concerned with repetition rate.
- g. Alexander's energy level is preposterously high.

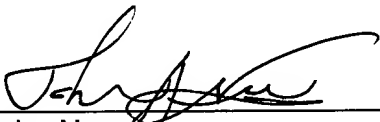
Conclusion

35. Thus, based on the above, Ihlemann and Alexander do not show the features of items 6 and 7 respectively, as presumed in the Office Action dated April 12, 2005.

36. There are various methods described to achieve sufficient fluence on or in material by concentrating energy as described in USPN 5,656,186, alternative to focusing, lens and/or Gaussian.

37. I have carefully reviewed the Response to the Office Action prepared and filed herewith in the present application, and hereby verify that, to the best of my information and belief, the factual assertions set forth therein are correct and complete, and I am also in complete agreement as to the opinions expressed therein.

38. All statements made above of my own knowledge are true, that all statements made above on information and belief are believed to be true, and that these statements were made with the knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under Title 18 United States Code Section 1001, and may jeopardize the validity of above-identified application or any patent issuing therefrom.



John Nees

9/8/2005

Date

CURRICULUM VITAE

JOHN A. NEES

Citizenship: U.S.A.

Education

M.S., Optics The Institute of Optics, University of Rochester, Rochester, NY, 1985.

B.S., Physics Kansas State University, 1983.

Professional Experience

Associate Research Scientist, Center for Ultrafast Optical Science, University of Michigan, Ann Arbor, Michigan, November 2001 to present.

Assistant Research Scientist, Center for Ultrafast Optical Science, University of Michigan, Ann Arbor, Michigan , November 1995-November 2001.

Senior Associate Research Engineer, Ultrafast Science Laboratory, University of Michigan, Ann Arbor, Michigan , September 1988 - November 1995.

Laboratory Engineer, Laboratory for Laser Energetics, University of Rochester, Rochester, New York, September 1985 - August 1988.

Research Assistant, Laboratory for Laser Energetics, University of Rochester, Rochester, New York, August 1984 - September 1985.

Engineer in Training, United Technologies Research Center, East Hartford, Connecticut, January - August 1984.

Research Interests

John Nees is currently the director of the λ^3 relativistic laser laboratory at the Center for Ultrafast Optical Science. His research is aimed at making precision studies of Relativistic Optics including ultrafast x-ray sources, attosecond physics, high-harmonic generation, and electron and ion acceleration. One application of this work is the possible production of positrons with sub-picosecond synchronization. He also collaborates with groups involved in laser-based materials growth and micromachining.

Professional responsibilities

Director of the relativistic lambda cubed lab at the University of Michigan:

Responsibilities include managing graduate students, post doctoral scholars, and visiting research scientists to develop applications of extremely intense light. This includes daily lab meetings, weekly group meetings, preparation of publications, and conference presentations, reporting of research results to funding agencies and contract monitors, preparation and evaluation of research proposals and securing intellectual property.

Consulting

Huron Valley Steel Corp: Laser based materials analysis

Picometrix Inc: Optoelectronic instrumentation prototyping

International Collaborators:

Ferenz Krausz, Director of the Max Plank Institute for Quantum Electronics, Germany: attosecond science

Daniel Kaplan, former director of research at Thomson CSF, France, currently COE of Fastlite, streak camera technology, photoconductive switching, active phase control of femtosecond pulses

Pierre Turnois formerly of Thomson CSF, currently of Fastlite, France: devices for chirped pulse amplification, active phase control of femtosecond pulses

Jean-Claude Kieffer, director of the Petawatt laser project INRS, Canada: laser-based x-rays and high-harmonic generation

Shinichi Wakana of Fujitsu Laboratories, Japan: Electro-optic and photoconductive switches and sub-micron ultrafast circuit testing

Shuntaro Watanabe Professor of Toklyo University: wavefront control for enhancement of nonlinearities using ultrafast pulses

Sadhiro Takuma, former director of Kansai Research Establishment: High saturation fluence laser development

Hajime Nishioka, professor of University for Electro Communications, Japan: ultrasonic damage detection and Yb-base lasers. Patric George and Frederic Druon, Optics Institute France: Yb-based laser materials development

Nam, Korea: Carrier-Envelope phase stabilization.

Selected Publications

“Phase-matched enhancements of high-harmonic soft X-rays by adaptive wave-front control with a genetic algorithm,” D. Yoshitomi, J. Nees, N. Miyamoto, T. Sekikawa, T. Kanai, G. Mourou, S. Watanabe, Appl. Phys. B-Lasers Opt., **78**, 275 (4 March, 2004).

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“Isolated attosecond pulses generated by relativistic effects in a wavelength-cubed focal volume,” N. M. Naumova, J. A. Nees, B. Hou, G. A. Mourou, and I. V. Sokolov, Opt. Lett. **29**, 778 (2004).

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“High resolution hard x-ray spectroscopy of femtosecond laser-produced plasmas with a CZT detector,” L-M. Chen, P. Forget, R. Toth, J-C Kieffer, A. Krol, C.C. Chamberlain, B. Hou, J. Nees, G. Mourou., Rev. Scientific Instr., **74**, 12, 5035-8 (Dec. 2003).

“Direct measurement of the photoelectric response time of bacteriorhodopsin via electro-optic sampling,” Xu, J. (Dept. of Electr. Eng. & Comput. Sci., Michigan Univ., Ann Arbor, MI, USA); Stickrath, A.B.; Bhattacharya, P.; Nees, J.; Varo, G.; Hillebrecht, J.R.; Ren, L.; Birge, R.R. Source: Biophysical Journal, v **85**, n 2, Aug. 2003, p 1128-34.

“Electron acceleration by few-cycle laser pulses with single-wavelength spot size,” Dudnikova, G.I. (Inst. of Comput. Technol., Novosibirsk, Russia); Bychenkov, V.Yu.; Maksimchuk, A.; Mourou, G.; Nees, J.; Bochkarev, S.G.; Vshivkov, V.A. Source:

Physical Review E (Statistical, Nonlinear, and Soft Matter Physics), v **67**, n 2, Feb. 2003, p 26416-1-7

- "Multi-diagnostic comparison of femtosecond and nanosecond pulsed laser plasmas," Zhang, Z. (Dept. of Electr. Eng. & Comput. Sci., Michigan Univ., Ann Arbor, MI, USA); VanRompay, P.A.; Nees, J.A.; Pronko, P.P. Source: Journal of Applied Physics, v **92**, n 5, 1 Sept. 2002, p 2867-74.
- "Directly diode-pumped Yb : KY(WO₄)(2) regenerative amplifiers" H. H. Liu, J. Nees G. Mourou, Opt. Lett. **27**, 9, 722-4 (1 May 2002).
- "Yb:KGd(WO₄)₂ chirped-pulse regenerative amplifiers," H. Liu, J. Nees, G. Mourou, S. Biswal, G.J. Spuhler, U. Keller, N. V. Kuleshov Optics-Communications. **203**, 3-6, 15 March 2002, p.315-21.
- "Epitaxial SnO₂ thin films grown on (1012) sapphire by femtosecond pulsed laser deposition," J. E. Dominguez, X. Q. Pan, L. Fu, P. A. Van Rompay, Z. Zhang, J. A. Nees, P. P. Pronko, Journal of App. Phys. **91**, no.3; 1 Feb. 2002; p.1060-5
- "On the design of experiments for the study of relativistic nonlinear optics in the limit of single-cycle pulse duration and single-wavelength spot size," G. Mourou, Z. Chang, A. Maksimchuk, J. Nees, S.V. Bulanov, N.M. Naumova, Vyu. Bychenkov, T.Zh. Esirkepov, F. Pegoraro, H. Ruhl, Plasma-Physics-Reports. **28**, 1, Jan. 2002; p.12-27(Translated from: Fizika-Plazmy, **28**, 1, Jan. 2002, p.14-31).
- "12-mJ, 350-fs Yb:GdCOB regenerative amplifier," F. Druon, F.Balembois, P. Georges, A. Brun, Seung Whan Bahk, J. Nees, G. Mourou, G. Cheriaux, J-P. Chambaret, G. Aka, D. Vivien Optics Comm. vol.**199**, no.1-4; 15 Nov. 2001; p.181-7
- "Diode-pumped Kerr-lens mode-locked Yb:KY(WO₄)₂ laser," H. Liu, J. Nees, G. Mourou, Optics-Letters. **26**, no.21; 1 Nov. 2001; p.1723-5.
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- "Isotope enrichment in laser-ablation plumes and commensurately deposited thin films," P.P. Pronko, P. A. VanRompay, Z. Zhang, and J. Nees, Phys. Rev. Lett. **83**, 2596-2599 (1999).
- "Ultrafast ytterbium-doped bulk lasers and laser amplifiers," C. Honninger, R. Paschotta, M. Graf, F. Morier-Genoud, G. Zhang, M. Moser, S. Biswal, J. Nees, A. Braun, G. A. Mourou, I. Johannsen, A. Giesen, W. Seeber, U. Keller, Appl. Phys. B-Lasers Opt. **69**, 1, 3-17 (July 1999)
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- "Ytterbium-doped glass regenerative chirped-pulse amplifier," S. Biswal, J. Nees, A. Nishimura, H. Takuma, G. Mourou, Opt. Commun. **160**, 92-7 (3 January 1999)
- "Wave-front correction of femtosecond terawatt lasers by deformable mirrors," F. Druon, G. Cheriaux, J. Faure, J. Nees, M. Nantel, A. Maksimchuk, J-C Chanteloup, G. Vdovin, Opt. Lett. **23**, 13, 1043-5 (1 July, 1998)

- "Ensuring compactness, reliability, and scalability for the next generation of high-field laser," J. Nees, S. Biswal, F. Druon, M. Nantel, G. Mourou, A. Nishimura, H. Takuma, J. Itatani, J. C. Chanteloup, and C. Hönninger, *IEEE Selected Topics in Quantum Electronics*, **4**, 2 March/April 1998, 376-84
- "Efficient energy extraction below the saturation fluence in a low-gain low-loss regenerative chirped-pulse amplifier," S. Biswal, J. Itatani, J. Nees, G. A. Mourou, *IEEE Selected Topics in Quantum Electronics*, **4**, 2 March/April 1998 421-5.
- "Temporal contrast in ti:sapphire lasers: characterization and control," M. Nantel, J. Itatani, A.-C. Tien, J. Faure, D. Kaplan, M. Bouvier, T. Buma, P. VanRompay, J. Nees, P. P. Pronko, D. Umstader, and G. Mourou, *IEEE Selected Topics in Quantum Electronics*, **4**, 2 March/April 1998.
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- "A fiber-mounted, micromachined photoconductive probe with 15 nV/Hz(1/2) sensitivity" R. K. Lai, J. R. Hwang, J. Nees, T. B. Norris, J. F. Whitaker, *Appl. Phys. Lett.* **69**, 13, 1843-5 9 (23 September 1996).
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- "Photoconductive sampling probe with 2.3 ps temporal resolution and 4- μ V sensitivity", J. Kim, S. Williamson, J. Nees, S. Wakana, and John Whitaker, *Appl. Phys. Lett.* **62**, 18, (3 May 1993).
- "1.4 ps Risetime High-Voltage Photoconductive Switching," T. Motet, J. Nees, S. Williamson, G. Mourou, *Appl. Phys. Lett.*, **59**, 1455-1457, (1991).
- "100 GHz Traveling-Wave Electro-Optic Phase Modulator," J. Nees, G. Mourou, S. Williamson, *Appl. Phys. Lett.*, **54**, 1962, (1989).
- "Novel Method for Ultrahigh-Frequency Electro-Optic Time-Domain Reflectometry," T. Jackson, J. Nees, R. Vallee, and G. Mourou, *Elect. Lett.* **23**, 1130-1131 (October 1987).
- "Noncontact Electro-Optic Sampling with a GaAs Injection Laser," J. Nees and G. Mourou, *Electron. Lett.* **22**, 918-919 (August 1986).

Patents

"Electro-Optic Measurement (Network Analysis) System," #4,745,361, May 17, 1988.

"Scanning Probe Having Picosecond Temporal and Nanometer Spatial Resolution," #5,442,300, August 15 1995.

"Selectively Triggered High-Contrast Laser", #5,541,947,1996.

"Method for Laser Induced Isotope Enrichment." # 6,586,696 July 1, 2003.

Scientific Service and Distinctions

Internal funding review for University of Michigan.

NSF proposal review panels: Biophysics, Sept. 1999, Lasers and LEDs March 2002, March 2003, and March 2004.

JASON committee review, July 2001.

Papers reviewed for Optics Lett., JOSA B, and Appl. Phys. Lett. , J. Appl. Phys..

Invited speaker at Ultrafast Lasers for the Canadian Light Source, Michigan Economic Development Corporation, and The European Atto-Network Symposium, International Conference on UltraIntense Lasers.

ED-200 and Laser Precision Corp. RJP 735). Ablation depths and surface roughnesses were measured by a Dektak 3030 Auto II stylus profilometer and by optical microscopes. The morphology of the ablation holes was investigated by scanning electron microscopy (Zeiss DSM 962).

2 Results

2.1 308 nm (33 ns)

Polished samples (arithmetic average roughness $R_a = 2$ nm) show an ablation threshold of about 20 J/cm^2 .

Table 1. Experimental details of the ablation set-up

Wave-length	Pulse duration	Repetition rate	Mask aperture	Focal length of imaging lens/numerical aperture ^a	Demagnification ratio	Ambient air pressure
308 nm	33 ns	5 Hz	3.2 mm	100 mm/0.1	10:1	1 bar
248 nm	28 ns	5 Hz	3.2 mm	100 mm/0.1	10:1	1 bar
193 nm	22 ns	5 Hz	2.0 mm	100 mm/0.1	10:1	1 bar
248 nm	500 fs	5 Hz	6.0 mm	150 mm/0.1	50:1	10^{-2} mbar ^b

^a Large focal length/low numerical aperture result in similar fluence at front and rear position of a 1 mm thick sample

^b To prevent air breakdown in the focus in front of the sample surface

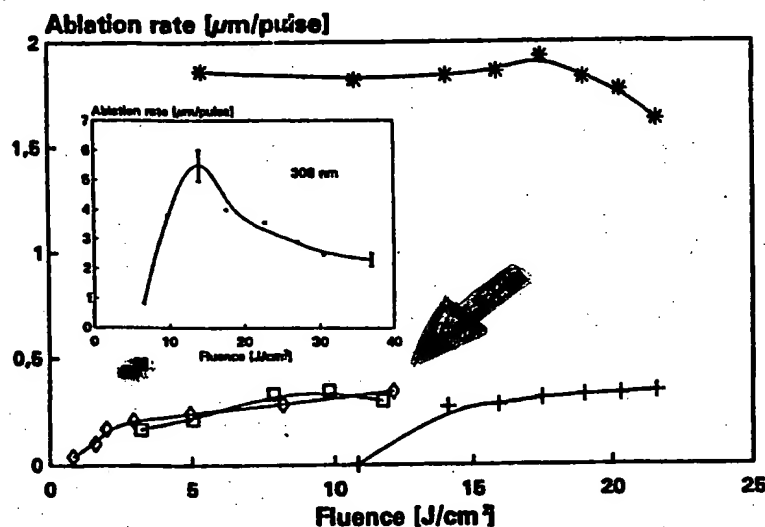


Fig. 1. Ablation rates of fused silica measured at different wavelengths and pulse durations.
 • 248 nm (valid for rough samples from the beginning on and for polished samples during phase 2); + 248 nm (phase 1);
 ■ 193 nm (rear side); □ 193 nm (front side);
 ◇ 248 nm (fs-pulses)

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